

FLUIDIZED-BED DEVELOPMENT AT JPL

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Silicon deposition on silicon seed particles by silane pyrolysis in a fluidized bed reactor (FBR) has been under investigation as a low-cost, high throughput method to produce high purity polysilicon for solar cell applications. The emphasis of the research at JPL is fundamental understanding of fluidized bed silicon deposition. The mechanisms involved were modeled as a six-path process: (1) Heterogeneous deposition; (2) Homogeneous decomposition; (3) Coalescence; (4) Coagulation; (5) Scavenging; and (6) Chemical vapor deposition growth on fines.

INTRODUCTION

The Flat-Plate Solar Array (FSA) Project was initiated in 1975 by the Department of Energy (DOE, then ERDA) to develop the technology for low-cost terrestrial photovoltaic arrays. In silicon material area, the Project was assigned a responsibility to develop the capability of producing polycrystalline silicon of semiconductor grade purity, suitable for the manufacture of terrestrial solar cells, at a price of less than \$14/Kg (in 1980 \$)(ref.1,2). In 1976, the Silicon Material Task of FSA initiated the fluidized bed silicon deposition in-house work parallel to the contractual development work by Union Carbide Corporation via silane decomposition and by Battelle via zinc reduction of silicon tetrachloride.(ref.3) Initially, the JPL in-house work was primarily installed to keep the contract management on top of the cutting-edge technologies that JPL/FSA was managing for DOE. Over the years, the emphasis has been redirected to fundamental understanding of silicon production in the advanced reactors and to identify critical research areas in order to complement the industrial process development efforts. Based on the simple chemistry of silane decomposition, the reactor technology studies at JPL in-house included: free space reactor, direct conversion to molten silicon, and fluidized bed silicon deposition. Constrained by funding situation in the contractual development work, FSA/Si Material Task decided to concentrate on the fluidized bed reactor (FBR) for low-cost production of silicon. Complemented by the JPL FBR research, Union Carbide Corporation has developed the essential parts of this technology in a R&D scale under a FSA contract. Currently, DOE considers the R&D of FBR technology mature enough to

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be pursued by the industry for final pilot plant development leading to commercialization. Consequently, the JPL in-house FBR development was concluded in June 1985, whereas the overall FSA silicon material work was completed in December 1985. This paper summarizes the fluidized bed development at JPL, and reports the engineering and scientific status of JPL FBR with a brief recommendation for the remaining R&D.

REACTION MECHANISM

The mechanisms involved were modeled as a six-path process (ref. 4):

Deposition and Decomposition: In a FBR, silane is thermally decomposed and silicon is formed via two major paths (see Figure 1); 1) heterogeneous chemical vapor deposition on the surface of seed particles in the emulsion phase of the bed, and 2) homogeneous gas phase pyrolysis forming condensable Si species in the bubble phase of the bed leading to formation of nuclei.

Coalescence: The tiny silicon nuclei move about within the bubble by Brownian motion and fuse by the action of Van der Waals forces - the path (3) in Figure 1. The growth rate of a nucleus with a radius r is found to be

$$\frac{dr}{dt} = \left(\frac{2kTf_s}{3\pi\mu} \right) \frac{1}{r^2} \quad (1)$$

where k is the Boltzmann constant

μ is the viscosity of nucleus in the gas stream

f_s is the volume fraction of solids in the gas-solids mixture

T is temperature in $^{\circ}\text{K}$.

By the time nuclei grow to $0.3\text{ }\mu\text{m}$ size, they are so few and far between that the growth rate drops by a factor of 10^{-4} . The resulting fine particle from this stage of nuclei coalescence is called coccal silicon. As a result of bubble distortion and kneading of fluid eddies in the fluidization medium, these coccal silicon fines may come out of the gas bubble into the emulsion phase.

Coagulation: Within the bubbles the coccal silicon fines coagulate into clusters. The size of a cluster grows as the residence time of the bubble in the bed increases. The resulting cluster is light in density, and travels with the gas bubble. At the top of the bed, bubbles burst and typically $<10\%$ of the silicon input in the forms of clusters and residual coccal silicon fines are elutriated and collected in the exit filter. Limited by the terminal velocity of the operation of fluidized bed, the maximum size of elutriated clusters is about $10\text{ }\mu\text{m}$ under the experimental conditions.

Scavenging: In the emulsion phase, the seeds sweep up the fine particles, which are coccal silicon and initial clusters released from the bubbles. This phenomenon is called scavenging.

Based on the data, it appears that in order to incorporate fines, the following criteria must be met:

- 1) The temperature of the scavenging region must be higher than the Tammann temperature* of silicon - defined as $0.52 \times$ (the melting point of silicon in °K).

This threshold temperature is about 610°C. In the FBR case, it represents the minimum seed particle surface temperature required to incorporate fines. As shown by SEM pictures in earlier work(ref.5), scavenging of fines at temperature below the Tammann temperature leads only to loose adhesion.

- 2) Chemical vapor deposition (CVD) reaction serves to cement the homogeneous fines into the growth. This surface adsorption of homogeneously nucleated particles and simultaneous heterogeneous deposition is similar to the model proposed in the carbon deposition literature.(ref.6.7)

Heterogeneous Growth of Fines: Fines, mostly coccal silicon, grow by heterogeneous chemical vapor deposition in the emulsion phase - i.e. the path (6) in Figure 1.

REACTOR DESIGN

The fluidized bed reactor is depicted in Figure 2. It is constructed from a 6-inch schedule 40 stainless steel 316 pipe and has dimensions of 6.065 inch inside diameter x 48 inch high. It has an expanded head of 24 inch x 24 inch to allow entrained particles to drop back into the bed.

Gas Distributor:

Five different types of gas distributors were investigated at JPL(ref.8) namely, a porous carbon unit with nine spouts, a nozzle with 1/4-inch diameter opening, a multi-layer screen (trade name - DYNAPORE) distributor and two-distributors fabricated from either one or two layers of No. 325 mesh stainless steel screen supported by a 1/20 inch thick plate perforated with 1/8 inch diameter holes. All these gas distributors were able to provide a bubbling fluidized bed with the exception of the nozzle-type distributor. Although a spouting distributor creates vigorous mixing of solids, it is difficult to ensure a firm contact of the metallic cooling coil underneath the porous carbon material. Distributors with one or two layers of No. 325 mesh screens were found most effective because of the simplicity in design to provide external water cooling.

A distributor temperature about 350°C will cause partial decomposition of silane at the distributor followed by clogging of the particles in the bed. Three distributor cooling systems were investigated: (1) Water cooling at the periphery of the distributor and a 1/2 inch thick cooling ring located

*Tammann temperature is an index for surface mobility or the minimum temperature at which a solid undergoes solid-solid interactions such as a transformation from a non-wetting to a wetting condition.

between the distributor and the reactor flange; (2) The same cooling system supplemented with internal water cooling tubes within the fluidized bed, located 1/4 inch above the distributor screen; and (3) water cooling at the periphery of the distributor in addition to a water cooling coil located beneath the distributor. A copper cooling coil was silver soldered to a 1/20 inch thick plate with multiple holes. The distributor screen is spot welded to the support plate to ensure firm contact. The first two cooling systems were inadequate to keep the distributor temperature below 350°C. The water cooling tubes located within the fluidized bed not only increased the load on the heaters, but also caused an excessive temperature gradient between the fluidized bed and the reactor wall resulting in silicon deposits on the wall. The third cooling system was found most satisfactory, both to keep the distributor temperature below 350°C and to eliminate wall deposits.

Heaters: The reactor was externally heated by a two-zone fast response, 12 inch high silicon carbide heater with a total output of 9 kw. Both zones are independently controlled in order to achieve an isothermal condition in the bed with 3 inches from the distributor. On the top of silicon carbide heater there is a single-zone independently controlled 18 inches high ceramic clam-shell heater with a heating element made of an alloy of iron, chromium, cobalt, and aluminum with a total power output of 9 kw. The main purpose of this heater is to provide a thermal insulation around the reactor and distribute the heat input throughout the bed height.

Product Withdrawal System

The fluidized bed is equipped with a continuous silicon withdrawal system to maintain a constant bed height.(ref.9) This system is depicted in Figure 3. It consists of an externally water-cooled silicon withdrawal tube, 15 inches long x 0.87 inches I.D. The particles in the withdrawal tube were fluidized at around minimal fluidization velocity in order to: (1) keep silane out of the withdrawal tube, (2) permit accumulation of large particles in the tube, and (3) cool the tube more effectively by preventing a sudden drop of hot bed particles within the tube.

At the bottom of the tube, an electronically controlled pinch valve (trade name - RED VALVE) is located. This valve is opened at a predetermined frequency (10-15 second intervals) for a duration of one second to allow particles to drop into a tee. The tube is water-cooled and provides cooling for the particles. The particles in the tee are pneumatically transported to a cyclone separator and collected in a silicon holding tank.

Reactor Liner System

The quartz liner design is depicted in Figure 4. It consists of a 5.75-in. outside diameter x 4 feet long quartz tube with 1/8-in. wall thickness. The top end of quartz liner is supported against a ring with 1/4-in. groove and a graphoil gasket. The bottom end of the quartz liner stands on a piston-gas distributor assembly with 1/4-in. groove and a quartz sleeve used as a gasket. The outside surface of piston consists of a pneumatic cylinder. The interface is vacuum and pressure sealed with a viton o-ring. The entire assembly is water cooled to maintain integrity of the o-ring. The pneumatic cylinder is mounted against the reactor flange.

Initially, the reactor containing quartz liner is filled with silicon particles. The reactor is closed and evacuated to remove oxygen prior to heating. The particles are fluidized with nitrogen or helium. A pressure of 200 psig. is applied at the pneumatic cylinder to mechanically seal the top end of quartz liner against the graphoil gasket. The pressure at the pneumatic cylinder compensates the differential thermal expansion between the stainless steel reactor wall and the quartz liner in axial direction. The bottom end of the quartz liner is not completely sealed and hydrogen is permitted to flow at a rate of less than 1 SLPM. During entire heating cycle, as well as silicon deposition experiment, a differential positive pressure of 20-in. water column is maintained between annular space and inlet port for silane/hydrogen gas mixture. This procedure assures that hydrogen can leak into the reactor but under these conditions silane/hydrogen mixture cannot leak into the annular space and cause wall deposits.

Seed Generation Device

A jet milling technique to grind silicon by impinging jets has been studied in this work for FBR seed particle generation. This device involves grinding of silicon particles by impingement of two opposing nitrogen jets carrying silicon particles. It is lined with high-density polypropylene and polyurethane to avoid exposure of silicon to metallic surfaces. It has an impact chamber of 0.75 in. diameter x 2 in. long. It is equipped with two opposing nitrogen jets with orifice diameter of 0.078 inches.

Silicon Cleaning Device

In the silicon cleaning device (ref.10), silicon particles are fluidized by the cleaning solution. The entire system in contact with either silicon or acid is constructed out of polyethylene and polypropylene. It consists of three plastic bottles of 3.5 inches diameter x 8 inches high. Each bottle holds up 500 grams of silicon. The bottom of the bottle is equipped with a 50 μ m pore diameter plastic screen and distributor to provide a tangential flow of cleaning solution for good mixing. The top of the bottle is equipped with another 75 μ m pore diameter screen to retain the particles within the bottles. All these three bottles are installed in a plastic tank which is filled with up to 2 gallons of cleaning solution. The cleaning solution is circulated through the system via a plastic pump. The flow rate of cleaning solution is adjusted by a plastic needle valve to obtain desired fluidization of silicon particles.

The cleaning of silicon seed obtained from grinding of silicon chunks includes the following steps:

- (1) Deionized water wash in a liquid-solid fluidized bed mode to remove fine silicon particles of less than 75 μ m in diameter.
- (2) Seed cleaning in a mixture of two parts of 12N HCl and one part of 16N HNO₃ in fluidized bed mode for 20 minutes.
- (3) Acids are drained and particles are washed with deionized water in a fluidized bed mode until the effluent water is neutral.
- (4) Etching of silicon particles with 48% HF in a fluidized bed mode for 20 minutes.

- (5) HF is drained and particles are washed with deionized water in a fluidized bed mode until effluent water is neutral and has a resistivity of 16 mega-ohms.
- (6) The wet particles are dried in a diffusion furnace at 150°C under nitrogen blanket.
- (7) After drying, particles are transferred into a plastic bag and sealed.

REACTOR PERFORMANCE

The overall mass balance data indicate that more than 90% of the total silicon fed into the reactor is deposited on the silicon seed particles and the remaining 10% ends up as elutriated fines. The rate of silicon production is a function of silane concentration, seed particle size and U/U_{mf} . Figure 5 shows that in the 6 inch FBR, a silicon production rate of 1.5 Kg/hr at 30% silane concentration or 3.5 Kg/hr at 80% silane concentration is achievable without formation of excessive fines. Most of the experiments conducted at JPL of the 6 inch FBR were of short duration (~4 hrs).

Silicon Particle Growth

The large seed particles (e.g., more than 200 μm diameter) in a FBR sweep up and collect the homogeneously nucleated fines (e.g., smaller than 1 μm) onto the particle surfaces, followed by sealing of the simultaneous heterogeneous deposition. Consider a seed particle of radius r and density ρ travelling up and down in a fluidized bed. The surrounding environment contains homogeneously nucleated fines of weight concentration ω_f travelling at a velocity U' of the fluidizing gas. The mass balance around a particle due to the scavenging and incorporation of homogeneously nucleated fines is (Ref. 11).

$$\rho 4\pi r^2 \frac{dr}{dt} = \pi r^2 (\Delta U) \omega_f \quad (2)$$

where ΔU can be approximated by the actual fluidized gas velocity U' . ω_f can be approximated from the silane concentration and the subsequent concentration reduction in the coalescence step leading to the fines.

$$\omega_f = C_{Ao} \times M_{Si} \times \frac{\text{Kg}}{K_S + \text{Kg}} \times \frac{1}{1 + X_A} \times 0.5 \times \left(\frac{25 \times 10^{-10}}{0.15 \times 10^{-6}} \right)^2 \quad (3)$$

silane	Si mol.	homo	dilution	Average	Number
feed	weight	reaction	factor	in the	of fines
conc.		fraction	due to	bed	reduced
			hydrogen		from collisions
			formation		of nuclei (50Å)
					to fines (0.3 μm)

where X_A is the fraction of silane concentration in the feed.

K_g is the first order rate constant for homogeneous decomposition of silane, 10.4 sec^{-1} at 650°C . (ref.12)

K_s is first order rate constant for heterogeneous decomposition of silane, 3.1 sec^{-1} at 650°C . (ref.13)

On the other hand, the seed particle growth due to heterogeneous deposition can be described by the following mass balance:

$$N \cdot 4\pi r^2 \frac{dr}{dt} = \frac{K_s}{K_s + K_g} C_{Ao} M_{Si} uA \quad (4)$$

where:

$$N = \frac{\omega_o}{\frac{4}{3} \pi r_o^3 \rho} \quad (5)$$

N is the number of particles in the bed,

ω_o is the initial bed weight

r_o is radius of initial seed particle

u is the superficial velocity of the gas, $\text{cm}^3/\text{cm}^2 \cdot \text{sec}$

The total growth rate of a particle equals the sum of equations (2) and (4).

Based on equations (2) and (4), the theoretical particle growth can be estimated as shown in Table 1. The actual particle growth (averaged) can be obtained from a mass balance over the experiment, together with the measured particle size distribution of the product. It can be seen that the calculated values agree nicely with the experimentally measured sizes.

Combining equations (4) and (5), the following relationship holds:

$$\frac{dr}{dt} = p \frac{r_o F}{\omega_o} C_{Ao} \quad (6)$$

where p is a constant, $\frac{1}{3} \left(\frac{K_s}{K_s + K_g} \right) \rho M_{Si}$

F is the total flow rate, cm^3/sec .

This means that the deposition rate is linearly proportional to C_{A0} , r_0 and F , and is inversely proportional to ω_0 . If r_0 , and F and ω_0 are held constant, a simple correlation of deposition rate (dr/dt) versus silane feed concentration (C_{A0}) is obtained. This can be shown by normalizing the deposition rate data, $\mu\text{m/hr}$, from Table 1 versus initial particle size of $227 \mu\text{m}$, initial bed weight of 400.2 moles of silicon and total flow rate of 3.0 moles/min. These rate data (dr/dt) are plotted against percentage feed silane concentration in hydrogen (X_A) in Figure 6. This results in a straight line correlation. In this way, the rate constant of the overall growth model, K , is obtained as $13 \mu\text{m/hr}$ for the 6-in. JPL FBR design.

$$\left. \frac{dr}{dt} \right|_{r_0, \omega_0, F} = K X_A \quad (7)$$

PRODUCT PURITY

The purity of FBR product was determined for two cases: (1) the FBR was operated without a liner and silicon particles were exposed to hot stainless steel reactor wall, and (2) the FBR was operated with a quartz liner to isolate the silicon particles from stainless steel reactor wall.

For the first case, the metallic impurities in FBR product is shown in Table 2. It indicates that silicon product in that case is heavily contaminated with metallic impurities. The iron, chromium and nickel impurities are approximately 70%, 17% and 12% of total identified impurities which are similar to the composition of stainless steel 316 material. Thus, it is evident that hot silicon particles erode the stainless steel wall material and incorporate metallic impurities on top of the seed particles.

For the second case, the metallic impurities in FBR product are shown in Table 3 (ref. 10). Within the accuracy limit of the analytical method, e.g., in this case, spark source mass spectroscopy is selected as the best analytical method for most of metallic impurities. The results suggest that the FBR processing does not introduce contamination into the product, and the quartz liner is effective to preserve the seed particle purity.

RECOMMENDATIONS

Although the JPL in-house research program has contributed over the years towards a fundamental understanding of fluidized bed silicon deposition, the following problems are still left to be solved.

- Demonstrate that FBR product particles are of semiconductor-grade purity (i.e., The particulate form of product makes the current analytical techniques difficult to reach to less than ppb for some of the impurities).
- Eliminate the problem of small particle accumulation in the FBR.

- Develop fluid jet milling to generate FBR seeds in a continuous loop with the FBR process.
- Demonstrate the operation of FBR in a steady state long-duration fashion.

CONCLUSION

This paper summarizes the fluidized-bed development at JPL, and characterizes the JPL FBR in terms of engineering design parameters such as gas distributor, cooling systems, heaters, product withdrawal, and a quartz liner with support. Also, the operation support systems including silicon seed generation and seed cleaning device are described. The overall mass-balance data indicate that more than 90% of the total silicon fed into the reactor is deposited on seed particles and the remaining less-than 10% of the total silicon bed ends up as the fines elutriants. A six-inch diameter FBR was demonstrated to be capable of producing 3.5 Kg/hr of silicon using 80% silane feed concentration, with a first order linear growth rate constant at 650°C of 13 $\mu\text{m/hr}$. The benefit of a quartz liner in avoiding the erosion contamination from fluidizing silicon particles against the metallic reactor wall was demonstrated with product purity experiments.

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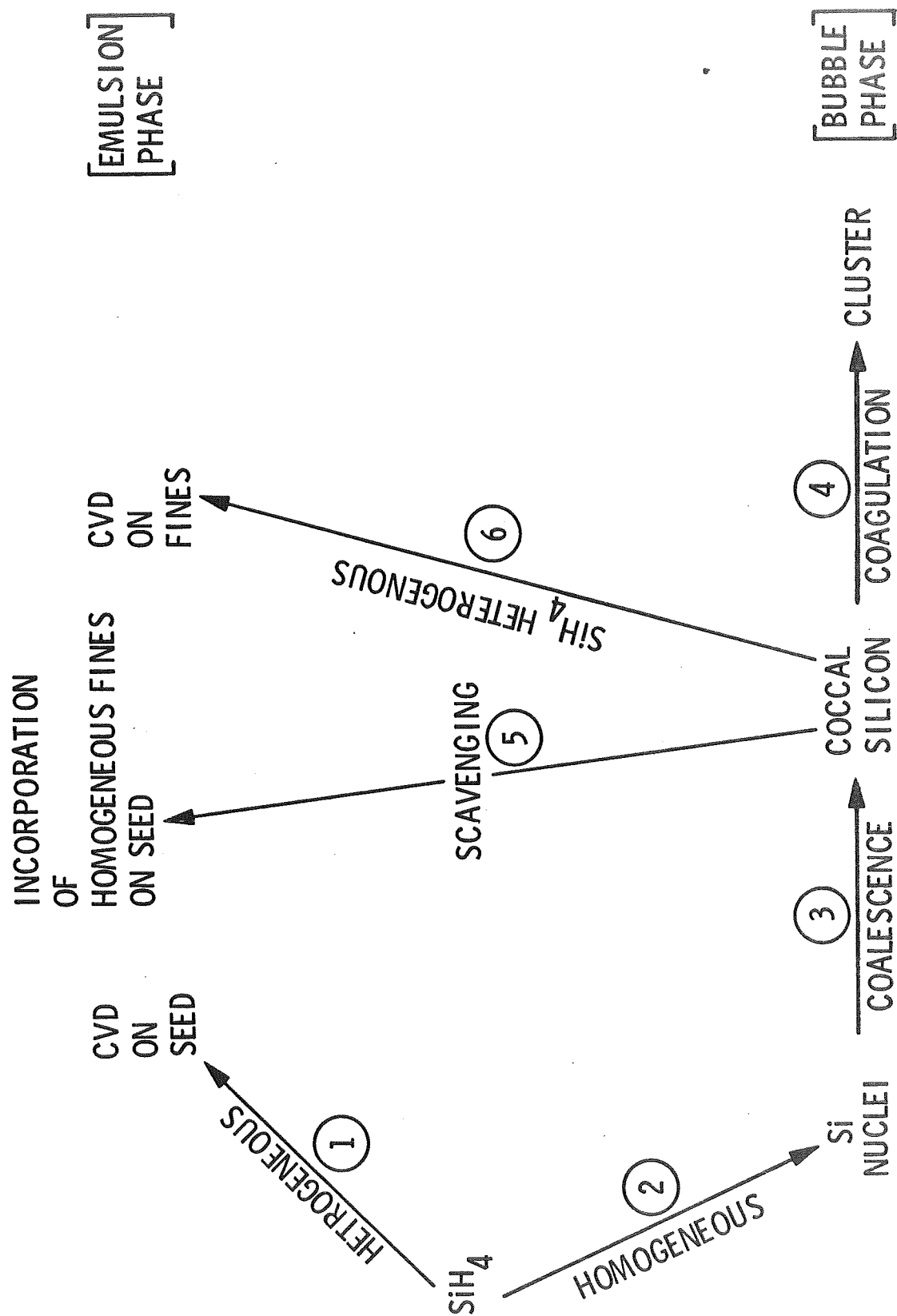


Figure 1. Reaction Paths for Silane Pyrolysis in a FBR

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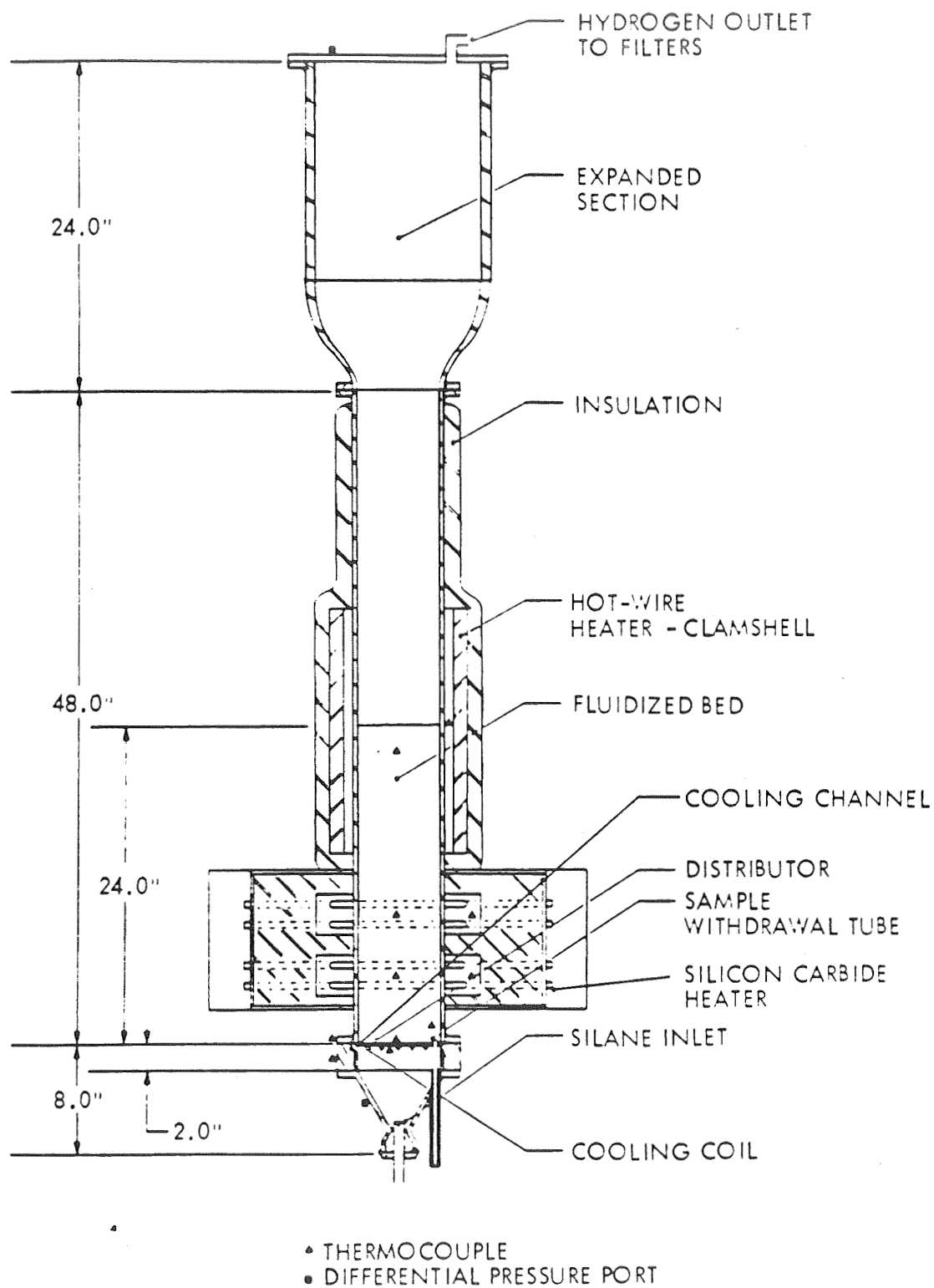


Figure 2. Six-inch Diameter Fluidized Bed Reactor

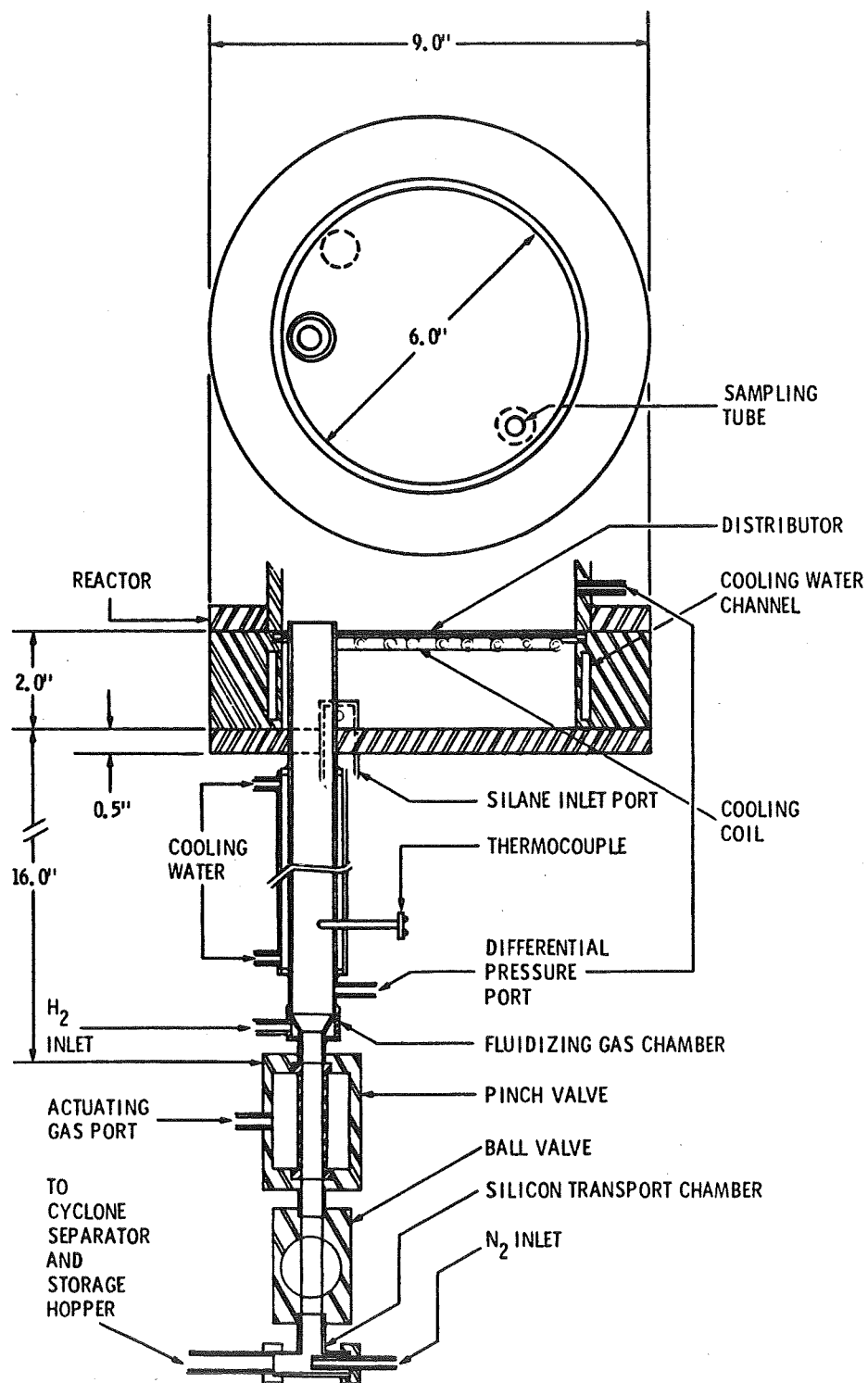
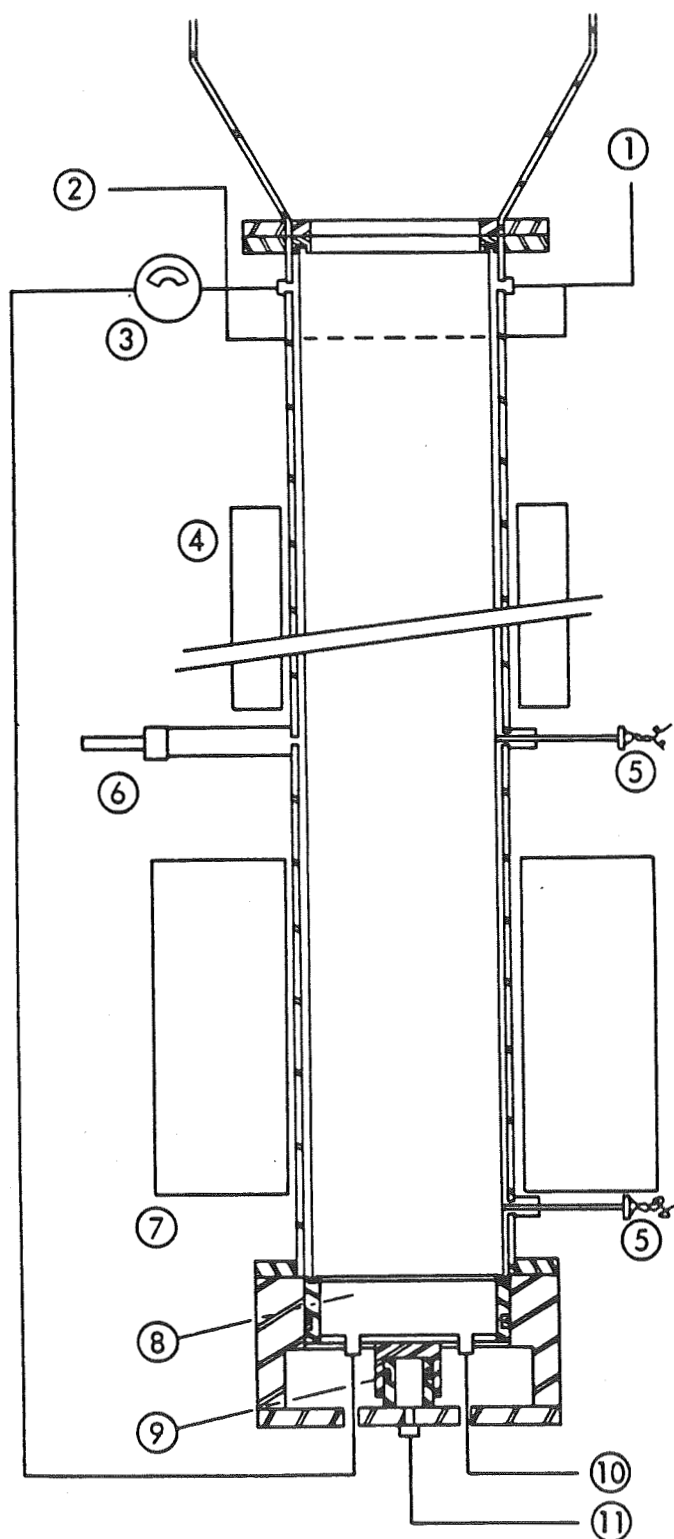


Figure 3. Product Withdrawal System



- ① HYDROGEN EXHAUST
- ② HYDROGEN INLET
- ③ DIFFERENTIAL PRESSURE GAGE
- ④ CLAMSHELL HEATER
- ⑤ THERMOCOUPLE
- ⑥ PYROMETER
- ⑦ SILICON CARBIDE HEATER
- ⑧ PISTON
- ⑨ PNEUMATIC CYLINDER
- ⑩ SILANE INLET
- ⑪ NITROGEN INLET

Figure 4. Quartz Liner for FBR

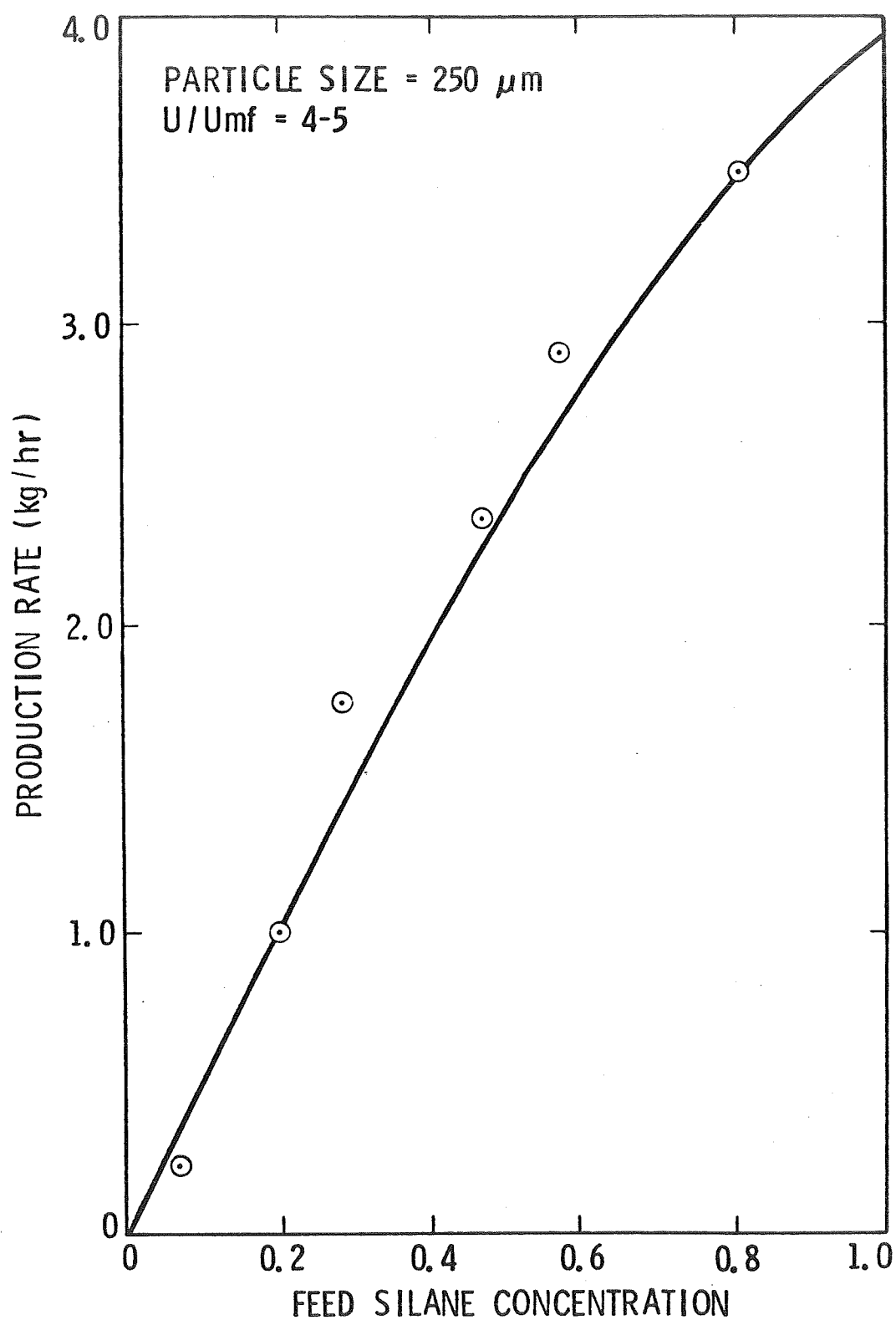


Figure 5. Experimental Production Rate vs. Silane Concentration

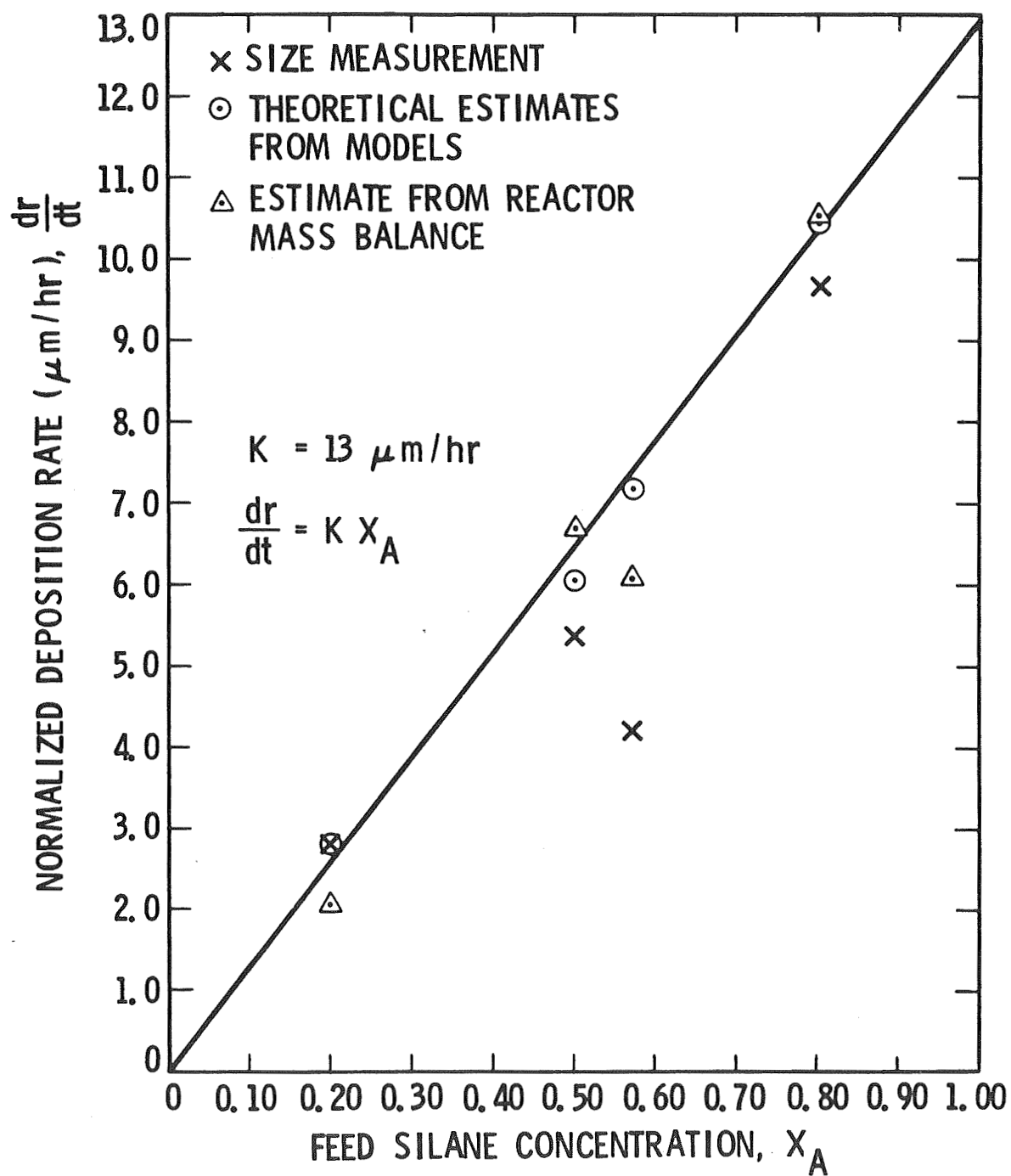


Figure 6. Normalized Deposition Rate vs. Feed Silane Concentration

Table 1. Silicon Particle Growth

SILANE CONCENTRATION (VOL %)	RUN DURATION (MIN.)	INITIAL DIAMETER OF PARTICLE (μm)	THEORETICAL ESTIMATE FOR DEPOSITION DUE TO SCAVENGING* (μm)	THEORETICAL ESTIMATE FOR HETEROGENEOUS DEPOSITION** (μm)	THEORETICAL FROM MODELS		MEASURED FINAL DIAMETER SIZE ANALYSIS (μm)	ESTIMATED FROM MASS BALANCE	
					DIAM. (μm)	% DEV. FROM MEAS.		DIAM. (μm)	% DEV. FROM MEAS.
20	90	227	5.4	3.0	235.4	0%	235.5	233.2	-1%
50	120	268	17.4	16.0	301.4	+1.3%	297.6	305.1	+2.5%
57	120	236	17.0	10.0	263.0	+4.4%	251.9	259.0	+2.8%
80	173	212	18.0	14.0	244.0	+1.0%	241.5	244.2	+1.1%

AVERAGE INITIAL BED WEIGHT: 10 KG; $T = 650^\circ\text{C}$, $U/U_{MF} = 5$

* EQUATION (2)

** EQUATION (3)

Table 2. Purity of FBR Product without a Liner (PPmw), (Neutron Activation Analysis - Lawrence Livermore Lab)

JPL RUN No.	Cr	Fe	Ni	Co	Mo	W	Na	TOTAL IDENTIFIED
111 (SEED)	4		1		0.05	0.04	1.3	
111-4p*	16 (15%)	77 (72%)	11 (11%)	0.16	0.76 (0.7%)	0.1	2	107
111-8p**	45 (17%)	185 (70%)	31 (12%)	0.36	3.8 (1.4%)	0.15	1.3	266
115p***	46 (17%)	197 (71%)	30 (11%)	0.4	1 (0.4%)	0.1	1.1	275
115 INGOT - BOTTOM	0.7						1.3	
115 INGOT - MIDDLE	0.2						4.2	
115 INGOT - TOP	0.3						1.2	

REACTOR WALL MATERIAL S.S.316: Cr 17%, Fe 70%, Ni 12%, Mo 2.5%, Mn 2%, Si 1%
EXPERIMENTAL CONDITIONS FOR RUN #111 AND 115, REFER TO REPORT (26).

* 4P IS PRODUCT OBTAINED AFTER 4 hrs.

** 8P IS PRODUCT OBTAINED AFTER 8 hrs.

*** P IS FINAL SILICON PRODUCT

Table 3. Purity of FBR Product with Quartz Liner (PPma)

ELEMENTS	JET MILLED AND ACID CLEANED SEED FOR FBR	FBR PRODUCT*
	SSMS**	SSMS
P	0.2	0.1
Fe	≤ 0.6	≤ 0.6
Cr	0.03	< 0.02
Ni	< 0.5	< 0.5
Cu	≤ 0.02	< 0.02
Zn	≤ 0.04	< 0.04
Co	≤ 0.1	≤ 0.1
Mn	≤ 0.02	< 0.02
Na	≤ 0.1	≤ 0.1
Mg	< 1	< 1
Al	0.05	0.05
S	< 1	< 1
K	< 0.1	< 0.1
Ca	0.1	0.1

* RUN NOS. 502 AND 503

** SPARK SOURCE MASS SPECTROSCOPY CONDUCTED BY THE NORTHERN ANALYTICAL LAB

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DISCUSSION

SANJURJO: Perhaps I missed it, but I didn't see any boron concentration data in the analysis of the silicon obtained with the jetmill process. Can you comment on this?

HSU: I'm sorry, but we don't have those results.